```
Si1 O7 W2 O6 41.3(4) . . . 4_655 ?
W2 O7 W2 O6 174.1(6) 3_665 . . 4_655 ?
W2 O7 W2 O6 -91.5(6) 2_655 . . 4_655 ?
Si1 O7 W2 O6 -41.3(4) . . . . ?
W2 O7 W2 O6 91.5(6) 3_665 . . . ?
W2 O7 W2 O6 -174.1(6) 2_655 . . . ?
Si1 O7 W2 O8 -131.4(7) . . . . ?
W2 O7 W2 O8 1.4(8) 3_665 . . . ?
W2 O7 W2 O8 95.8(9) 2_655 . . . ?
Si1 O7 W2 O8 131.4(7) . . . 2_655 ?
W2 O7 W2 O8 -95.8(9) 3_665 . . 2_655 ?
W2 O7 W2 O8 -1.4(8) 2_655 . . 2_655 ?
N1 C3 C4 C5 -171(3) . . . . ?
C3 C4 C5 C6 -176(4) . . . . ?
N1 C7 C8 C9 -155(5) . . . 5 ?
N1 C7 C8 C9 155(5) . . . . ?
C7 C8 C9 C10 9(11) . . . . ?
C9 C8 C9 C10 -96(8) 5 . . . ?
C4 C3 N1 C3 -177(2) . . . 5 ?
C4 C3 N1 C7 56(5) . . . . ?
C4 C3 N1 C11 -65(5) . . . . ?
C8 C7 N1 C3 -61(3) . . . 5 ?
C8 C7 N1 C3 61(3) . . . . ?
C8 C7 N1 C11 180.000(13) . . . . ?
C3 N1 C11 C12 56(2) 5 . . . ?
C3 N1 C11 C12 -56(2) . . . . ?
C7 N1 C11 C12 179.999(10) . . . . ?
N1 C11 C12 C13 180.00(3) . . . . ?
C11 C12 C13 C14 180.002(14) . . . . ?
C11' C12' C13' C14' -115(12) . . . . ?

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  _platon_squeeze_void_average_y
  _platon_squeeze_void_average_z
  _platon_squeeze_void_volume
  _platon_squeeze_void_count_electrons
  _platon_squeeze_void_content
   1 -0.051 -0.038  0.527      2333       649 ' '
   2  0.000  0.000  0.876        20        10 ' '
   3  0.333  0.667  0.542        19        10 ' '
   4  0.667  0.333  0.209        19        10 ' '
_platon_squeeze_details
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;

_diffrn_measured_fraction_theta_max    0.997
_diffrn_reflns_theta_full              25.02
_diffrn_measured_fraction_theta_full   0.997
_refine_diff_density_max    1.568
_refine_diff_density_min   -1.150
_refine_diff_density_rms    0.174
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Si1 O5 W1 O2 174(4) . . . . ?
W1 O5 W1 O2 33(5) 5 . . . ?
Si1 O5 W1 O4 -41.6(10) . . . . ?
W1 O5 W1 O4 176.5(7) 5 . . . ?
Si1 O5 W1 O1 140.9(12) . . . . ?
W1 O5 W1 O1 -1.0(7) 5 . . . ?
Si1 O5 W1 O6 47.7(10) . . . . ?
W1 O5 W1 O6 -94.1(6) 5 . . . ?
Si1 O5 W1 O3 -131.0(10) . . . . ?
W1 O5 W1 O3 87.2(6) 5 . . . ?
O9 O9' W2 O9' -51(10) . . . 4_655 ?
O9' O9' W2 O9 51(10) 4_655 . . . ?
O9 O9' W2 O6 177(10) . . . 4_655 ?
O9' O9' W2 O6 -132.1(6) 4_655 . . 4_655 ?
O9 O9' W2 O6 -100(10) . . . . ?
O9' O9' W2 O6 -49.8(6) 4_655 . . . ?
O9 O9' W2 O8 -6(11) . . . . ?
O9' O9' W2 O8 44.9(9) 4_655 . . . ?
O9 O9' W2 O8 88(10) . . . 2_655 ?
O9' O9' W2 O8 138.7(9) 4_655 . . 2_655 ?
O9 O9' W2 O7 100(15) . . . . ?
O9' O9' W2 O7 151(13) 4_655 . . . ?
O9' O9 W2 O9' 81(20) . . . 4_655 ?
O9' O9 W2 O9' -81(20) 4_655 . . . ?
O9' O9 W2 O6 -84(9) 4_655 . . 4_655 ?
O9' O9 W2 O6 -3(10) . . . 4_655 ?
O9' O9 W2 O6 3(10) 4_655 . . . ?
O9' O9 W2 O6 84(10) . . . . ?
O9' O9 W2 O8 94(10) 4_655 . . . ?
O9' O9 W2 O8 175(10) . . . . ?
O9' O9 W2 O8 -175(10) 4_655 . . 2_655 ?
O9' O9 W2 O8 -94(10) . . . 2_655 ?
O9' O9 W2 O7 140(10) 4_655 . . . ?
O9' O9 W2 O7 -140(10) . . . . ?
W1 O6 W2 O9' -142(2) . . . 4_655 ?
W1 O6 W2 O9' -127(3) . . . . ?
W1 O6 W2 O9 -143(2) . . . . ?
W1 O6 W2 O6 -38.5(18) . . . 4_655 ?
W1 O6 W2 O8 120.5(17) . . . . ?
W1 O6 W2 O8 30(3) . . . 2_655 ?
W1 O6 W2 O7 48.9(16) . . . . ?
W2 O8 W2 O9' -180(3) 3_665 . . 4_655 ?
W2 O8 W2 O9' 166(2) 3_665 . . . ?
W2 O8 W2 O9 165(2) 3_665 . . . ?
W2 O8 W2 O6 -21(3) 3_665 . . 4_655 ?
W2 O8 W2 O6 -89.1(13) 3_665 . . . ?
W2 O8 W2 O8 68.3(15) 3_665 . . 2_655 ?
W2 O8 W2 O7 -2.0(11) 3_665 . . . ?
Si1 O7 W2 O9' -119(13) . . . 4_655 ?
W2 O7 W2 O9' 14(13) 3_665 . . 4_655 ?
W2 O7 W2 O9' 108(13) 2_655 . . 4_655 ?
Si1 O7 W2 O9' 119(13) . . . . ?
W2 O7 W2 O9' -108(13) 3_665 . . . ?
W2 O7 W2 O9' -14(13) 2_655 . . . ?
Si1 O7 W2 O9 180.00(2) . . . . ?
W2 O7 W2 O9 -47.2(5) 3_665 . . . ?
W2 O7 W2 O9 47.2(5) 2_655 . . . ?
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O9' O9 O9' W2 83(4) 4_655 . . . ?
W1 O5 Si1 O5 -49.2(17) 5 . . 3_665 ?
W1 O5 Si1 O5 179.6(3) . . . 3_665 ?
W1 O5 Si1 O5 -179.6(3) 5 . . 2_655 ?
W1 O5 Si1 O5 49.2(17) . . . 2_655 ?
W1 O5 Si1 O7 65.6(8) 5 . . . ?
W1 O5 Si1 O7 -65.6(8) . . . . ?
W2 O7 Si1 O5 60.000(5) 3_665 . . 3_665 ?
W2 O7 Si1 O5 180.000(3) . . . 3_665 ?
W2 O7 Si1 O5 -60.000(2) 2_655 . . 3_665 ?
W2 O7 Si1 O5 -60.000(5) 3_665 . . . ?
W2 O7 Si1 O5 60.000(3) . . . . ?
W2 O7 Si1 O5 180.000(2) 2_655 . . . ?
W2 O7 Si1 O5 180.000(5) 3_665 . . 2_655 ?
W2 O7 Si1 O5 -60.000(4) . . . 2_655 ?
W2 O7 Si1 O5 60.000(3) 2_655 . . 2_655 ?
O10 O10' P1 O3 126.2(6) . . . . ?
O10 O10' P1 O3 -126.2(6) . . . 5 ?
O10 O10' P1 C1 0.000(5) . . . . ?
O10' O10 P1 O3 -62.7(8) . . . . ?
O10' O10 P1 O3 62.7(8) . . . 5 ?
O10' O10 P1 C1 180.000(4) . . . . ?
W1 O3 P1 O10' 84(2) . . . . ?
W1 O3 P1 O10 124.1(15) . . . . ?
W1 O3 P1 O3 -5(2) . . . 5 ?
W1 O3 P1 C1 -116.4(15) . . . . ?
C2 C1 P1 O10' 180.000(7) . . . . ?
C2 C1 P1 O10 180.000(3) . . . . ?
C2 C1 P1 O3 56.7(7) . . . . ?
C2 C1 P1 O3 -56.7(7) . . . 5 ?
W1 O4 W1 O2 -117.7(19) 4_655 . . . ?
W1 O4 W1 O1 75(3) 4_655 . . . ?
W1 O4 W1 O6 -18.8(19) 4_655 . . . ?
W1 O4 W1 O3 146.4(19) 4_655 . . . ?
W1 O4 W1 O5 67.8(19) 4_655 . . . ?
W1 O1 W1 O2 -173.7(10) 5 . . . ?
W1 O1 W1 O4 -6(3) 5 . . . ?
W1 O1 W1 O6 87.0(10) 5 . . . ?
W1 O1 W1 O3 -78.3(10) 5 . . . ?
W1 O1 W1 O5 1.2(8) 5 . . . ?
W2 O6 W1 O2 137.1(16) . . . . ?
W2 O6 W1 O4 35.3(17) . . . . ?
W2 O6 W1 O1 -123.8(16) . . . . ?
W2 O6 W1 O3 -46(3) . . . . ?
W2 O6 W1 O5 -50.3(16) . . . . ?
P1 O3 W1 O2 132.6(14) . . . . ?
P1 O3 W1 O4 -125.6(14) . . . . ?
P1 O3 W1 O1 34.4(14) . . . . ?
P1 O3 W1 O6 -45(3) . . . . ?
P1 O3 W1 O5 -39.9(13) . . . . ?
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C3 N1 C3 106(4) 5 . ?
C3 N1 C7 114(2) 5 . ?
C3 N1 C7 114(2) . . ?
C3 N1 C11 106(3) 5 . ?
C3 N1 C11 106(3) . . ?
C7 N1 C11 110(5) . . ?
C12 C11 N1 120(7) . . ?
C12 C11 H11A 107.4 . . ?
N1 C11 H11A 107.4 . . ?
C12 C11 H11B 107.4 . . ?
N1 C11 H11B 107.4 . . ?
H11A C11 H11B 106.9 . . ?
C11 C12 C13 123(9) . . ?
C11 C12 H12A 106.6 . . ?
C13 C12 H12A 106.6 . . ?
C11 C12 H12B 106.6 . . ?
C13 C12 H12B 106.6 . . ?
H12A C12 H12B 106.6 . . ?
C14 C13 C12 120(10) . . ?
C14 C13 H13A 107.4 . . ?
C12 C13 H13A 107.4 . . ?
C14 C13 H13B 107.4 . . ?
C12 C13 H13B 107.4 . . ?
H13A C13 H13B 107.0 . . ?
C13 C14 H14A 109.5 . . ?
C13 C14 H14B 109.5 . . ?
H14A C14 H14B 109.5 . . ?
C13 C14 H14C 109.5 . . ?
H14A C14 H14C 109.5 . . ?
H14B C14 H14C 109.5 . . ?
C12' C11' H11C 108.0 . . ?
C12' C11' H11D 108.0 . . ?
H11C C11' H11D 107.2 . . ?
C11' C12' C13' 110(3) . . ?
C11' C12' H12C 109.7 . . ?
C13' C12' H12C 109.7 . . ?
C11' C12' H12D 109.7 . . ?
C13' C12' H12D 109.7 . . ?
H12C C12' H12D 108.2 . . ?
C14' C13' C12' 113(3) . . ?
C14' C13' H13C 109.1 . . ?
C12' C13' H13C 109.0 . . ?
C14' C13' H13D 109.0 . . ?
C12' C13' H13D 109.0 . . ?
H13C C13' H13D 107.8 . . ?
C13' C14' H14D 109.5 . . ?
C13' C14' H14E 109.5 . . ?
H14D C14' H14E 109.5 . . ?
C13' C14' H14F 109.5 . . ?
H14D C14' H14F 109.5 . . ?
H14E C14' H14F 109.5 . . ?

loop_
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O9 W2 O7 163(3) . . ?
O6 W2 O7 87.0(6) 4_655 . ?
O6 W2 O7 87.0(6) . . ?
O8 W2 O7 71.6(7) . . ?
O8 W2 O7 71.6(7) 2_655 . ?
C4 C3 N1 112(2) . . ?
C4 C3 H3A 109.1 . . ?
N1 C3 H3A 109.1 . . ?
C4 C3 H3B 109.1 . . ?
N1 C3 H3B 109.1 . . ?
H3A C3 H3B 107.9 . . ?
C3 C4 C5 110(3) . . ?
C3 C4 H4A 109.7 . . ?
C5 C4 H4A 109.7 . . ?
C3 C4 H4B 109.7 . . ?
C5 C4 H4B 109.7 . . ?
H4A C4 H4B 108.2 . . ?
C6 C5 C4 113(4) . . ?
C6 C5 H5A 108.9 . . ?
C4 C5 H5A 108.9 . . ?
C6 C5 H5B 108.9 . . ?
C4 C5 H5B 108.9 . . ?
H5A C5 H5B 107.7 . . ?
C5 C6 H6A 109.5 . . ?
C5 C6 H6B 109.5 . . ?
H6A C6 H6B 109.5 . . ?
C5 C6 H6C 109.5 . . ?
H6A C6 H6C 109.5 . . ?
H6B C6 H6C 109.5 . . ?
N1 C7 C8 122(5) . . ?
N1 C7 H7A 106.9 . . ?
C8 C7 H7A 106.9 . . ?
N1 C7 H7B 106.9 . . ?
C8 C7 H7B 106.9 . . ?
H7A C7 H7B 106.7 . . ?
C7 C8 C9 124(6) . 5 ?
C7 C8 C9 124(6) . . ?
C9 C8 C9 41(7) 5 . ?
C7 C8 H8A 106.4 . . ?
C9 C8 H8A 129.4 5 . ?
C9 C8 H8A 106.4 . . ?
C7 C8 H8B 106.4 . . ?
C9 C8 H8B 67.3 5 . ?
C9 C8 H8B 106.4 . . ?
H8A C8 H8B 106.4 . . ?
C10 C9 C8 108(8) . . ?
C10 C9 H9A 110.0 . . ?
C8 C9 H9A 110.0 . . ?
C10 C9 H9B 110.0 . . ?
C8 C9 H9B 110.0 . . ?
H9A C9 H9B 108.4 . . ?
C9 C10 H10A 109.5 . . ?
C9 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C9 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
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O9' O9' W2 80(2) 4_655 . ?
O10' O10 P1 66.0(18) . . ?
O10 O10' P1 67(2) . . ?
O5 Si1 O5 113.2(5) 3_665 . ?
O5 Si1 O5 113.2(5) 3_665 2_655 ?
O5 Si1 O5 113.2(5) . 2_655 ?
O5 Si1 O7 105.5(7) 3_665 . ?
O5 Si1 O7 105.5(7) . . ?
O5 Si1 O7 105.5(7) 2_655 . ?
O10' P1 O10 47(2) . . ?
O10' P1 O3 89.1(14) . . ?
O10 P1 O3 114.7(8) . . ?
O10' P1 O3 89.1(14) . 5 ?
O10 P1 O3 114.7(8) . 5 ?
O3 P1 O3 107.7(12) . 5 ?
O10' P1 C1 155(2) . . ?
O10 P1 C1 108.9(16) . . ?
O3 P1 C1 105.1(9) . . ?
O3 P1 C1 105.1(9) 5 . ?
O2 W1 O4 101.8(8) . . ?
O2 W1 O1 98.6(8) . . ?
O4 W1 O1 159.1(8) . . ?
O2 W1 O6 98.9(7) . . ?
O4 W1 O6 89.1(7) . . ?
O1 W1 O6 92.0(7) . . ?
O2 W1 O3 96.0(7) . . ?
O4 W1 O3 88.5(7) . . ?
O1 W1 O3 85.0(7) . . ?
O6 W1 O3 165.0(6) . . ?
O2 W1 O5 170.8(7) . . ?
O4 W1 O5 85.5(7) . . ?
O1 W1 O5 73.7(6) . . ?
O6 W1 O5 86.5(6) . . ?
O3 W1 O5 78.6(6) . . ?
O9' W2 O9' 20(4) 4_655 . ?
O9' W2 O9 16(3) 4_655 . ?
O9' W2 O9 16(3) . . ?
O9' W2 O6 103.7(17) 4_655 4_655 ?
O9' W2 O6 90(3) . 4_655 ?
O9 W2 O6 106.0(18) . 4_655 ?
O9' W2 O6 90(3) 4_655 . ?
O9' W2 O6 103.7(17) . . ?
O9 W2 O6 106.0(18) . . ?
O6 W2 O6 82.4(8) 4_655 . ?
O9' W2 O8 97.2(18) 4_655 . ?
O9' W2 O8 112(3) . . ?
O9 W2 O8 96.5(18) . . ?
O6 W2 O8 157.4(7) 4_655 . ?
O6 W2 O8 89.2(8) . . ?
O9' W2 O8 112(3) 4_655 2_655 ?
O9' W2 O8 97.2(18) . 2_655 ?
O9 W2 O8 96.5(18) . 2_655 ?
O6 W2 O8 89.2(8) 4_655 2_655 ?
O6 W2 O8 157.4(7) . 2_655 ?
O8 W2 O8 90.8(13) . 2_655 ?
O9' W2 O7 168.6(18) 4_655 . ?
O9' W2 O7 168.6(18) . . ?
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C13 H13A 0.9900 . ?
C13 H13B 0.9900 . ?
C14 H14A 0.9800 . ?
C14 H14B 0.9800 . ?
C14 H14C 0.9800 . ?
C11' C12' 1.50(2) . ?
C11' H11C 0.9900 . ?
C11' H11D 0.9900 . ?
C12' C13' 1.52(2) . ?
C12' H12C 0.9900 . ?
C12' H12D 0.9900 . ?
C13' C14' 1.48(3) . ?
C13' H13C 0.9900 . ?
C13' H13D 0.9900 . ?
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C14' H14E 0.9800 . ?
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P1 C1 H1A 108.9 . . ?
C2 C1 H1B 108.9 . . ?
P1 C1 H1B 108.9 . . ?
H1A C1 H1B 107.8 . . ?
C1 C2 H2A 109.5 . . ?
C1 C2 H2B 109.5 . . ?
H2A C2 H2B 109.5 . . ?
C1 C2 H2C 109.5 . . ?
H2A C2 H2C 109.5 . . ?
H2B C2 H2C 109.5 . . ?
W1 O1 W1 116.6(10) 5 . ?
P1 O3 W1 136.4(9) . . ?
W1 O4 W1 144.9(11) . 4_655 ?
Si1 O5 W1 125.3(5) . 5 ?
Si1 O5 W1 125.3(5) . . ?
W1 O5 W1 96.1(6) 5 . ?
W2 O6 W1 151.7(8) . . ?
Si1 O7 W2 122.3(6) . 3_665 ?
Si1 O7 W2 122.3(6) . . ?
W2 O7 W2 94.1(8) 3_665 . ?
Si1 O7 W2 122.3(6) . 2_655 ?
W2 O7 W2 94.1(8) 3_665 2_655 ?
W2 O7 W2 94.1(8) . 2_655 ?
W2 O8 W2 122.7(11) . 3_665 ?
O9' O9 O9' 80(10) 4_655 . ?
O9' O9 W2 81(4) 4_655 . ?
O9' O9 W2 81(4) . . ?
O9 O9' O9' 50(10) . 4_655 ?
O9 O9' W2 83(4) . . ?
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O6 W1 1.906(13) . ?
O7 Si1 1.74(3) . ?
O7 W2 2.277(15) 3_665 ?
O7 W2 2.277(15) . ?
O7 W2 2.277(15) 2_655 ?
O8 W2 1.899(10) . ?
O8 W2 1.899(10) 3_665 ?
O9 O9' 0.46(9) 4_655 ?
O9 O9' 0.46(9) . ?
O9 W2 1.698(19) . ?
O9' O9' 0.59(12) 4_655 ?
O9' W2 1.691(18) . ?
O10 O10' 1.17(5) . ?
O10 P1 1.50(3) . ?
O10' P1 1.478(19) . ?
Si1 O5 1.683(17) 3_665 ?
Si1 O5 1.683(17) 2_655 ?
P1 O3 1.569(15) 5 ?
W2 O9' 1.691(19) 4_655 ?
W2 O6 1.886(14) 4_655 ?
W2 O8 1.899(10) 2_655 ?
C3 C4 1.503(19) . ?
C3 N1 1.509(18) . ?
C3 H3A 0.9900 . ?
C3 H3B 0.9900 . ?
C4 C5 1.529(19) . ?
C4 H4A 0.9900 . ?
C4 H4B 0.9900 . ?
C5 C6 1.48(3) . ?
C5 H5A 0.9900 . ?
C5 H5B 0.9900 . ?
C6 H6A 0.9800 . ?
C6 H6B 0.9800 . ?
C6 H6C 0.9800 . ?
C7 N1 1.521(19) . ?
C7 C8 1.51(2) . ?
C7 H7A 0.9900 . ?
C7 H7B 0.9900 . ?
C8 C9 1.52(2) 5 ?
C8 C9 1.52(2) . ?
C8 H8A 0.9900 . ?
C8 H8B 0.9900 . ?
C9 C10 1.45(3) . ?
C9 H9A 0.9900 . ?
C9 H9B 0.9900 . ?
C10 H10A 0.9800 . ?
C10 H10B 0.9800 . ?
C10 H10C 0.9800 . ?
N1 C3 1.509(18) 5 ?
N1 C11 1.51(2) . ?
C11 C12 1.50(2) . ?
C11 H11A 0.9900 . ?
C11 H11B 0.9900 . ?
C12 C13 1.52(2) . ?
C12 H12A 0.9900 . ?
C12 H12B 0.9900 . ?
C13 C14 1.51(3) . ?
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H14E H 1.0590 0.4249 0.5321 0.294 Uiso 0.20 1 calc PR . -2
H14F H 1.1441 0.4799 0.5501 0.294 Uiso 0.20 1 calc PR . -2

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 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
Si1 0.032(3) 0.032(3) 0.028(5) 0.000 0.000 0.0160(16)
P1 0.061(6) 0.080(5) 0.091(6) 0.012(2) 0.025(5) 0.031(3)
W1 0.0547(5) 0.0535(5) 0.0764(6) -0.0015(5) 0.0006(5) 0.0170(4)
W2 0.0784(8) 0.0783(10) 0.0545(7) -0.0188(7) -0.0094(4) 0.0391(5)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters are
only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

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C1 C2 1.50(3) . ?
C1 P1 1.81(4) . ?
C1 H1A 0.9900 . ?
C1 H1B 0.9900 . ?
C2 H2A 0.9800 . ?
C2 H2B 0.9800 . ?
C2 H2C 0.9800 . ?
O1 W1 1.908(10) 5 ?
O1 W1 1.908(10) . ?
O2 W1 1.730(15) . ?
O3 P1 1.569(15) . ?
O3 W1 1.983(14) . ?
O4 W1 1.904(6) . ?
O4 W1 1.904(6) 4_655 ?
O5 Si1 1.683(17) . ?
O5 W1 2.183(11) 5 ?
O5 W1 2.183(11) . ?
O6 W2 1.886(14) . ?
```

```
O10 O 0.8058(15) 0.4029(8) 0.2903(12) 0.042(6) Uiso 0.60 2 d SP . .
O10' O 0.7762(12) 0.3881(6) 0.337(2) 0.064(14) Uiso 0.40 2 d SPD . .
Si1 Si 0.6667 0.3333 0.4719(5) 0.031(2) Uani 1 6 d S . .
P1 P 0.8583(5) 0.4292(3) 0.3419(4) 0.079(3) Uani 1 2 d SD . .
W1 W 0.83565(5) 0.49615(5) 0.46856(6) 0.0660(3) Uani 1 1 d . . .
W2 W 0.72027(4) 0.44054(8) 0.60371(6) 0.0704(5) Uani 1 2 d SD . .
C3 C 0.948(2) 0.5320(19) 0.6258(16) 0.149(14) Uiso 1 1 d D . .
H3A H 0.9521 0.5313 0.5819 0.179 Uiso 1 1 calc R A 1
H3B H 0.8945 0.5047 0.6362 0.179 Uiso 1 1 calc R A 1
C4 C 0.982(2) 0.6113(18) 0.6466(17) 0.146(13) Uiso 1 1 d D . .
H4A H 1.0329 0.6411 0.6305 0.176 Uiso 1 1 calc R . .
H4B H 0.9848 0.6131 0.6905 0.176 Uiso 1 1 calc R . .
C5 C 0.934(3) 0.644(3) 0.6254(19) 0.181(17) Uiso 1 1 d D . .
H5A H 0.9282 0.6384 0.5817 0.217 Uiso 1 1 calc R . .
H5B H 0.8841 0.6158 0.6435 0.217 Uiso 1 1 calc R . .
C6 C 0.966(3) 0.724(3) 0.641(3) 0.25(3) Uiso 1 1 d D . .
H6A H 0.9337 0.7421 0.6256 0.371 Uiso 1 1 calc R . .
H6B H 1.0157 0.7531 0.6223 0.371 Uiso 1 1 calc R . .
H6C H 0.9711 0.7304 0.6839 0.371 Uiso 1 1 calc R . .
C7 C 0.986(3) 0.4930(16) 0.7209(16) 0.149(18) Uiso 1 2 d SDU . .
H7A H 1.0147 0.5457 0.7336 0.178 Uiso 0.50 1 calc PR B 1
H7B H 1.0147 0.4690 0.7336 0.178 Uiso 0.50 1 calc PR B 1
C8 C 0.914(3) 0.4572(17) 0.756(3) 0.21(2) Uiso 1 2 d SDU . .
H8A H 0.8775 0.4148 0.7323 0.252 Uiso 0.50 1 calc PR . .
H8B H 0.8973 0.4940 0.7577 0.252 Uiso 0.50 1 calc PR . .
C9 C 0.908(5) 0.428(5) 0.819(3) 0.20(3) Uiso 0.50 1 d PDU . -1
H9A H 0.8837 0.4485 0.8452 0.244 Uiso 0.50 1 calc PR . -1
H9B H 0.8774 0.3732 0.8197 0.244 Uiso 0.50 1 calc PR . -1
C10 C 0.983(6) 0.452(7) 0.841(5) 0.33(6) Uiso 0.50 1 d PDU . -1
H10A H 0.9846 0.4606 0.8841 0.499 Uiso 0.50 1 calc PR . -1
H10B H 1.0186 0.4976 0.8209 0.499 Uiso 0.50 1 calc PR . -1
H10C H 0.9947 0.4124 0.8331 0.499 Uiso 0.50 1 calc PR . -1
N1 N 0.986(2) 0.4928(12) 0.6532(15) 0.133(13) Uiso 0.60 2 d SPD . 1
C11 C 1.065(3) 0.5324(14) 0.630(4) 0.17(2) Uiso 0.60 2 d SPDU . 1
H11A H 1.0901 0.5834 0.6466 0.202 Uiso 0.30 1 calc PR . 1
H11B H 1.0901 0.5067 0.6466 0.202 Uiso 0.30 1 calc PR . 1
C12 C 1.079(6) 0.539(3) 0.564(4) 0.19(2) Uiso 0.60 2 d SPDU . 1
H12A H 1.0525 0.5646 0.5477 0.224 Uiso 0.30 1 calc PR . 1
H12B H 1.0525 0.4880 0.5477 0.224 Uiso 0.30 1 calc PR . 1
C13 C 1.156(6) 0.578(3) 0.537(5) 0.20(3) Uiso 0.60 2 d SPDU . 1
H13A H 1.1826 0.6297 0.5526 0.243 Uiso 0.30 1 calc PR . 1
H13B H 1.1826 0.5529 0.5526 0.243 Uiso 0.30 1 calc PR . 1
C14 C 1.165(10) 0.583(5) 0.470(5) 0.29(6) Uiso 0.60 2 d SPDU . 1
H14A H 1.1274 0.5919 0.4524 0.441 Uiso 0.30 1 calc PR . 1
H14B H 1.1589 0.5354 0.4551 0.441 Uiso 0.30 1 calc PR . 1
H14C H 1.2149 0.6233 0.4599 0.441 Uiso 0.30 1 calc PR . 1
N1' N 0.986(2) 0.4928(12) 0.6532(15) 0.133(13) Uiso 0.40 2 d SPD . 2
C11' C 1.068(3) 0.5340(14) 0.667(4) 0.19(3) Uiso 0.40 2 d SPDU . 2
H11C H 1.0787 0.5778 0.6921 0.231 Uiso 0.20 1 calc PR . 2
H11D H 1.0787 0.5009 0.6921 0.231 Uiso 0.20 1 calc PR . 2
C12' C 1.121(4) 0.5607(18) 0.616(6) 0.19(3) Uiso 0.40 2 d SPDU . 2
H12C H 1.1595 0.5459 0.6216 0.233 Uiso 0.20 1 calc PR . 2
H12D H 1.1469 0.6157 0.6133 0.233 Uiso 0.20 1 calc PR . 2
C13' C 1.079(7) 0.527(11) 0.558(4) 0.19(3) Uiso 0.20 1 d PDU . -2
H13C H 1.0249 0.4990 0.5670 0.226 Uiso 0.20 1 calc PR . -2
H13D H 1.0883 0.5675 0.5303 0.226 Uiso 0.20 1 calc PR . -2
C14' C 1.101(13) 0.476(11) 0.530(7) 0.20(5) Uiso 0.20 1 d PDU . -2
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_refine_ls_weighting_details
 'calc w=1/[\s^2^(Fo^2^)+(0.0417P)^2^+504.4679P] where
P=(Fo^2^+2Fc^2^)/3'
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_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details
 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack    0.10(4)
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_refine_ls_number_parameters      130
_refine_ls_number_restraints      40
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_refine_ls_wR_factor_gt           0.1391
_refine_ls_goodness_of_fit_ref    0.982
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 _atom_site_fract_y
 _atom_site_fract_z
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 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_symmetry_multiplicity
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 _atom_site_disorder_group
C1 C 0.953(2) 0.4766(10) 0.3139(16) 0.102(12) Uiso 1 2 d SD . .
H1A H 0.9606 0.4417 0.2886 0.122 Uiso 0.50 1 calc PR . .
H1B H 0.9606 0.5189 0.2886 0.122 Uiso 0.50 1 calc PR . .
C2 C 1.011(2) 0.5053(11) 0.3624(16) 0.110(13) Uiso 1 2 d SD . .
H2A H 1.0605 0.5306 0.3447 0.165 Uiso 1 2 calc SR . .
H2B H 1.0046 0.4635 0.3869 0.165 Uiso 0.50 1 calc PR . .
H2C H 1.0042 0.5405 0.3872 0.165 Uiso 0.50 1 calc PR . .
O1 O 0.8892(11) 0.4446(5) 0.4813(8) 0.061(5) Uiso 1 2 d S . .
O2 O 0.9086(9) 0.5864(9) 0.4772(7) 0.086(5) Uiso 1 1 d . . .
O3 O 0.8531(8) 0.4877(8) 0.3829(6) 0.067(4) Uiso 1 1 d . . .
O4 O 0.7561(5) 0.5122(11) 0.4464(8) 0.061(5) Uiso 1 2 d S . .
O5 O 0.7570(9) 0.3785(5) 0.4519(7) 0.040(4) Uiso 1 2 d S . .
O6 O 0.7990(8) 0.4780(7) 0.5481(6) 0.059(4) Uiso 1 1 d . . .
O7 O 0.6667 0.3333 0.5495(12) 0.045(7) Uiso 1 6 d S . .
O8 O 0.7537(12) 0.3768(6) 0.6342(9) 0.074(6) Uiso 1 2 d S . .
O9 O 0.7508(16) 0.502(3) 0.661(2) 0.08(3) Uiso 0.50 2 d SPD . .
O9' O 0.743(3) 0.514(2) 0.649(2) 0.05(2) Uiso 0.25 1 d PD . .
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_diffrn_radiation_monochromator  '10cm confocal mirrors'
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'Bruker-Nonius APEX II CCD camera on \k-goniostat'
_diffrn_measurement_method       '\f & \w scans'
_diffrn_detector_area_resol_mean '4096x4096pixels / 62x62mm'
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_diffrn_reflns_limit_k_max        24
_diffrn_reflns_limit_l_min        -26
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'COLLECT (Hooft, R.W.W., 1998)'
_computing_cell_refinement
'DENZO (Otwinowski & Minor, 1997) & COLLECT (Hooft, R.W.W., 1998)'
#Although determined using DirAx, the cell is refined in the HKL
#package during data reduction
_computing_data_reduction
'DENZO (Otwinowski & Minor, 1997) & COLLECT (Hooft, R.W.W., 1998)'
_computing_structure_solution     'SHELXS-97 (Sheldrick, 1997)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics     'Ortep-3 for Windows (Farrugia,
1997)'
_computing_publication_material   'WinGX (Farrugia, 1999)'

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;
 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR
and
 goodness of fit S are based on F^2^, conventional R-factors R are
based
 on F, with F set to zero for negative F^2^. The threshold expression
of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc.
and is
 not relevant to the choice of reflections for refinement.  R-factors
based
 on F^2^ are statistically about twice as large as those based on F,
and R-
 factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef  Fsqd
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 '-x+y, -x, z'
 '-x+y, y, z'
 'x, x-y, z'
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 'x+2/3, y+1/3, z+1/3'
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 '-x+y+2/3, y+1/3, z+1/3'
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 '-y+1/3, x-y+2/3, z+2/3'
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 'x+1/3, x-y+2/3, z+2/3'
 '-y+1/3, -x+2/3, z+2/3'

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_cell_length_c                    22.471(5)
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_cell_angle_beta                  90.000(5)
_cell_angle_gamma                 120.000(5)
_cell_volume                      8359(3)
_cell_formula_units_Z             3
_cell_measurement_temperature     120(2)
_cell_measurement_reflns_used     45479
_cell_measurement_theta_min       2.91
_cell_measurement_theta_max       27.48

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_exptl_absorpt_coefficient_mu     9.364
_exptl_absorpt_correction_type    Multi-scan
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2007)'

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data_2008src1078_r3m

_publ_section_comment
;
The NBu4 cation had disordered carbon chains (C7 to C10) and (C11 to
C14), due to this refinement was carried out
with all the atoms within the NBu4 cations isotropic. The positions of
these atoms could not be resolved
absolutely, the highly disordered groups causing some intra and
intermolecular contacts involving these groups to
be short.
A H+ ion must be present in the structure to balance the charge, this
is most likely coordinating between the P=O
groups at the top of the anion, however it can not be seen due to the
quality of the data.
Squeeze was carried out on the structure, to make allowance for
disordered solvent molecules.
;

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;
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 'C54 H124 N3 O37 P3 Si W9'
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 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
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 'Si'  'Si'   0.0817   0.0704
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'P'  'P'   0.1023   0.0942
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'W'  'W'  -0.8490   6.8722
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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loop_
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